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IS 1448-126 (1988): Methods of test for petroleum and its products, Part 126: Determination of ash content in raw and calcined petroleum coke [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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IS : 1448 [P : 126] - 1988
Adopted : 9 May 1988
(Reaffirmed 2006)

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 126]

DETERMINATION OF ASH CONTENT IN RAW AND CALCINED PETROLEUM COKE

(First Reprint MAY 1999)
UDC 665·777·4 : 543·822

Adapted from ASTM D 4422-84

1. SCOPE

1.1 This Indian Standard covers the determination of the ash content of petroleum coke.

2. OUTLINE OF METHOD

2.1 A representative sample of petroleum coke is dried, ground and ashed in a muffle furnace at 700 to 750°C. The residue or ash is expressed as a percentage of the dry petroleum coke.

3. SIGNIFICANCE AND USE

3.1 The ash content is one of the properties used to evaluate petroleum coke and indicates the amount of undesirable residue present. Acceptable ash content varies with the intended use.

4. INTERFERENCES

4.1 High sulphur content of furnace gases, regardless of the source of the sulphur, may react with an alkaline ash to produce erratic results. The furnace shall be swept with air to achieve oxidation and to decrease the sulphur content of the gases.

4.2 Preparation and testing of the sample for analysis shall neither remove nor add ash. Improper dividing, sieving and crushing equipment and some muffle furnace lining material may contaminate the sample.

5. APPARATUS

5.1 Crucibles — Low wide form glazed porcelain or platinum, 30 ml capacity.

NOTE — For further analysis on ash, only platinum crucibles shall be used.

5.2 Muffle Furnace — With temperature control between 700 and 750°C and equipped with a means to regulate air circulation.

5.3 Analytical Balance — Capable of weighing up to 0·1 mg.

5.4 Drying Oven — Controlled at 110 ± 5°C.

5.5 Desiccator

6. PREPARATION OF APPARATUS

6.1 The muffle furnace, when initially set up, shall be tested for adequate air circulation. The air flow is adequate if replicate samples do not produce a lower ash at higher air flow rates with the same furnace loading. Maintain air flow at the same level for subsequent analysis to ensure consistency in analytical technique.

7. SAMPLE PREPARATION

7.1 Crush the bulk sample to pass 6·7 mm sieve. If quantity exceeds 2·5 kg divide the sample to obtain 2·5 kg and crush this fraction to pass 850 micron sieve. Further divide the sample to obtain a portion of approximately 200 g and crush to pass a 250-micron IS Sieve. This is the analytical sample which is dried to constant mass at 110 ± 5°C (*see* Note).

NOTE — If the bulk sample appears wet it shall be air dried prior to crushing, to avoid caking.

8. PROCEDURE

8.1 Weigh into a coded, pre-ignited crucible, 10 ± 0·1 mg of the dried analysis sample passing through 250-micron IS sieve.

8.2 Place the crucible into a cold muffle furnace. Heat directly above 700°C until constant mass (± 0·2 mg) is obtained. Do not exceed 750°C.

8.3 Allow the furnace to cool to 150°C and transfer the crucible to the desiccator for futher cooling.

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8.4 Weigh the crucible and ash to the nearest 0·1 mg.

9. CALCULATION

9.1 Calculate the ash percent in the analysis sample as follows:

$$\text{Ash, percent} = \frac{A-B}{C} \times 100$$

where

A = mass in g of crucible and residue,
B = mass in g of empty crucible, and
C = mass in g of the sample for analysis.

10. REPORT

10.1 Report the ash content as mass percent to the third decimal (thousandth of a percent) when the average of replicate values permits.

10.2 Report the ash content as mass percent to the second decimal (hundredth of a percent) when only single values are determined.

11. PRECISION

11.1 Results of the duplicate tests shall not differ by more than the following limits:

Repeatability	*Reproducibility*
0·02	0·06